भारतीय मानक

Indian Standard

IS 18169 : 2023

मैग्नेशिया की औसत क्रिस्टल साइज ज्ञात करना

Determination of Average Crystal Size of Magnesia

ICS 81.080

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August 2023 **Price Group 4**

Refractories Sectional Committee, MTD 15

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Refractories Sectional Committee had been approved by the Metallurgical Engineering Division Council.

Periclase is cubic form of MgO with a perfect (001) cleavage. It is found in metamorphosed limestones and dolomites. Periclase has a structure similar to that of halite, NaCl, with magnesium and oxygen ions occupying the sites of sodium and chlorine respectively. Iron, zinc, nickel and manganese may substitute for magnesium in the natural mineral. Calcium, chromium and aluminum are also reported. Periclase derived from heating of dolomite is useful as a refractory material, for instance as a lining to blast furnaces. The color of periclase varies from white to yellow, or brown when iron is present. Small amounts of Cr and Fe produce green periclase and Mn-Zn-bearing periclase is grass-green in color. Periclase is a relatively high temperature mineral that occurs, for instance in marbles that result from the metamorphism of dolomites and magnesian limestones.

Slag corrosion resistance of sintered/fused magnesia is dependent on purity and size of periclase (MgO) crystals. When sintered/fused magnesia composite grains are polished properly and observed under optical microscope, rounded/rectangular grains of MgO (periclase) crystals of different sizes separated by very fine to thick grain boundaries are visible. Thickness of grain boundaries depends on the amount of impurities. When impurities content is more, rounded grains with thick grain boundaries are observed. When impurity content is very less, grain boundaries between periclase crystals are very fine. In highly crystalline magnesia (fused magnesia) periclase crystals of rectangular shape are observed.

In a sintered/fused magnesia specimen, periclase crystals of various sizes are observed. In order to determine average crystal size lineal intercept method is used. This standard prescribes the method of test for determination of average periclase crystal size of sintered or fused magnesia by optical microscope by using the lineal intercept method.

The composition of the Committee responsible for formulation of this standard is given in Annex A.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’.

Indian Standard

DETERMINATION OF AVERAGE CRYSTAL SIZE OF MAGNESIA

1 SCOPE

This standard prescribes the method of test for determination of average periclase (MgO) crystal size of sintered or fused magnesia by optical microscope by using the line intercept method.

2 TERMINOLOGY

For the purpose of this standard the following definitions shall apply.

2.1 Grain/Crystal — That area within the confines of the original (primary boundary), which can be revealed on a flat cross-section through the sample, polished and prepared for micrographic examination or that volume enclosed by the original (primary) boundary in the three-dimensional object.

2.2 Grain Intercept Count — Determination of the number of times a tests line cuts through individual grains on the plane-of-polish (tangent hits are considered as one half an interception; test lines that end within a grain are considered as on half an interception).

2.3 Graticule — A scale on glass or other transparent material placed in the eyepiece or at an intermediate plane on the optic axis of a light microscope for the location and measurement of objects.

2.4 Periclase Crystal Size — Average grain size of polycrystalline periclase (MgO) ceramic, determined from its microstructure using line intercept method.

3 SAMPLING

Depending upon the lot size, collect sufficient amount of material from randomly selected bags. Generally magnesia is supplied in 1 tonne jumbo bags. Collect 1 kg of material from each of the selected bags. Finally, take about 1 kg of material by coning and quartering and crush in a jaw crusher. Screen the material through a 5 mm and 2 mm sieve to get magnesia sample of size 2 mm to 5 mm. Take about 50 g of sample after coning and quartering. Wash the particles with clean water and dry for 24 h at 110 °C before mounting.

4 PROCEDURE

Periclase crystal can be observed and measured under reflected light microscope when one of the surfaces of magnesia particle is optically flat. For this, the following procedure is to be followed.

4.1 Mounting

Mount at least 10 particles or 20 particles in a 25 mm or 40 mm mould. For mounting, epoxy resin and hardener may be used. Number of particles to be analysed will be determined based on lot size. Minimum 20 particles shall be analysed for determination of average crystal size.

4.2 Polishing

Mounted magnesia grains are to be initially ground and polished until the surface is optically flat and grain boundaries are clearly visible when observed in reflected light under optical microscope.

4.3 Measurement of Crystal Size

4.3.1 Setup the optical microscope with 100x magnification with eye piece graticule. Place the polished sample on the stage of the reflected light optical microscope. Select one magnesia grain and adjust the focus to get clear and sharp image. Magnification is to be changed suitably to get at sufficient number of grains/crystals. If crystals are bigger magnification may be changed to 50x and in case of smaller crystals magnification may be changed to 200x.

4.3.2 Move the specimen until the grain boundary of one crystal matches with the end (zero position) of the graticule. Count the number of grain intercepts by the graticule and note the length of final intercept. The reading shall be recorded in a tabular form given below:

Sample holder No.	*Particle No.*	*No. of grain intercepts*	*Reading of final intercept in graticule divisions*	*Length of final intercept in µm*

4.3.3 Shift the sample to select another particle and then move slowly to match the end of the graticule with one of the grain boundaries. Count the number of grains and note the length of final intercept and record in the tabular form given in **4.3.2**.

4.3.4 Similarly count the grains and length of intercepts for remaining particles mounted in all the holders and record in tabular form given in **4.3.2**.

5 EXPRESSION OF RESULTS

5.1 Determine the average crystal size of the selected magnesia particle by using the following relationship:

$$\begin{aligned}&\text{Average Crystal size}(\mu\text{m})\\&= (\text{Length of the line } (\mu\text{m})/\text{Number of grains})\\&\quad\times \text{Multiplication Factor}\end{aligned}$$

NOTE — Use 1.5 as multiplication factor for periclase crystal

5.2 Similarly, calculate the average crystal size of all the particles.

5.3 Finally, calculate the overall average crystal size from the following relationship:

$$\begin{aligned}&Overall\ average\ crystal\ size\ (\mu m)\\&= \frac{\sum Average\ crystal\ size\ of\ all\ the\ particles\ (\mu m)}{Number\ of\ particles}\end{aligned}$$

6 TEST REPORT

The test report shall include at least the following information:

a) Lot size;
b) Number of grains observed;
c) Multiplication factor used;
d) Average periclase crystal size in µm; and
e) Overall average crystal size in µm.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Refractories Sectional Committee, MTD 15

Organization	*Representative(s)*
SKG Refractories Ltd, Kolkata	Dr Arup Kumar Chattopadhyay **(*Chairperson*)**
Calderys India Refractories Limited, Nagpur	Dr I. N. Chakraborty Shri Santanu Basak (*Alternate* I) Dr Saumen Sinha (*Alternate* II)
CSIR - Central Glass and Ceramic Research Institute, Kolkata	Dr H. S. Tripathi
Dalmia Institute of Science & Industrial Research, Sundargarh	Dr Prema Ranjan Rauta Shri Sudhir Kumar Panda (*Alternate* I) Shri Lalit Mohan Choudhury (*Alternate* II)
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IFGL Refractories Limited, Kalunga	Shri S. D. Majumdar Shri Sajahan Mandal (*Alternate*)
Indian Refractory Makers Association, Kolkata	Dr Arup Ghosh Shri Anirbandip Dasgupta (*Alternate*)
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OCL India Limited, Rajgangpur	Dr J. K. Sahu Dr A. K. Patnaik (*Alternate*)

Organization	*Representative(s)*
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Rashtriya Ispat Nigam Limited, Visakhapatnam	Shri S. K. Bhattacharya Shri P. S. Paul (*Alternate*)
Steel Authority of India Limited (SAIL), Bhilai Steel Plant, Bhilai	Shri Sandeep Srivastava Ms D. Satya Devi (*Alternate* I) Shri Amit Roy (*Alternate* II)
Steel Authority of India (SAIL), Centre for Engineering and Technology (CET), Ranchi	Shri Surendra Prasad Shri Aishwarya Dewangan (*Alternate*)
Steel Authority of India Limited (SAIL), Research & Development Centre for Iron & Steel (RDCIS), Ranchi	Shri M. K. Kujur Shrimati A. Bhattacharya (*Alternate*)
Tata Steel Limited, Kolkata	Shri Bajendra Shri Goutham Ghosh (*Alternate* I) Shri Prasanta Panigrahi (*Alternate* II)
TRL Krosaki Refractories Limited, Jharsuguda	Shri Arup Kumar Samanta Shri S. K. Subudhi (*Alternate* I) Shrimati Rajashree Swain (*Alternate* II)
In Personal Capacity (*Flat 1 A, Himkunj Cooperative Society, Block 1st Number – 167 B. P. Township, Kolkata*)	Dr Barun Deb Mukherjee
In Personal Capacity (*Plot No BC 224, Sector-1, Salt Lake, Kolkata*)	Shri K. K. Paul
BIS Directorate General	Shri Sanjiv Maini, Scientist 'F'/Senior Director and Head (Metallurgical Engineering) [Representing Director General (*Ex-officio*)]

Member Secretary
Saaqib Raahi
Scientist 'B'/Assistant Director
(Metallurgical Engineering), BIS

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